AA

PATENT ABSTRACTS OF JAPAN

(11)Publication number : 11-228245
(43)Date of publication of application : 24.08.1999

(51)Int.CI. C04B 37/02
B23K 35/30

(21)Application number : 10-052971
(22)Date of filing : 18.02.1998

(71)Applicant : NGK INSULATORS LTD
(72)Inventor : MAKINO TAKUMA
SHINKAI MASAYUKI

(54) BONDING COMPOSITION FOR BONDING DIFFERENT KINDS OF MEMBERS TO EACH OTHER, COMPOSITE MEMBER COMPRISING DIFFERENT KINDS OF MEMBERS BONDED WITH THE COMPOSITION, AND PRODUCTION OF THE COMPOSITE MEMBER

(57)Abstract:
PROBLEM TO BE SOLVED: To provide a bonding composition used for bonding two or more kinds of different members to each other, capable of bonding two or more kinds of the different members to each other in a sufficient bonding strength to produce a composite member not generating defects such as cracks on a member weak against thermal stresses among the different members, to provide the composite member comprising two or more kinds of the different members bonded to each other with the composition, and to provide a method for producing the composite member.
SOLUTION: This bonding composition for bonding different members to each other comprises a particulate substance for lowering thermal stresses and a hard brazing material based on a noble metal element. The composite member is obtained by bonding two or more kinds of the different members to each other with the composition or by filling a bonding site between the different members with the particulate substance for lowering the thermal stresses, pouring the molten hard brazing material into the bonding site and subsequently cooling the poured brazing material to bond the different members to each other.

LEGAL STATUS

[Date of request for examination]	04.08.1999
[Date of sending the examiner's decision of rejection]	05.02.2002
[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]	
[Date of final disposal for application]	
[Patent number]	3315919
[Date of registration]	07.06.2002
[Number of appeal against examiner's decision of rejection]	2002-04004
[Date of requesting appeal against examiner's decision of rejection]	07.03.2002
[Date of extinction of right]	

(19)日本国特許庁（ＪＰ）　(12)特　許　公　報（Ｂ２）　(11)特許番号
特許第3315919号
(P3315919)
(45)発行日　平成14年８月19日(2002.8.19)　(24)登録日　平成14年６月７日(2002.6.7)

(51)Int.Cl.[7]	識別記号	ＦＩ	
Ｃ０４Ｂ　37/02		Ｃ０４Ｂ　37/02	Ｂ
Ｂ２３Ｋ　35/30	３１０	Ｂ２３Ｋ　35/30	３１０Ｂ

請求項の数７(全　５　頁)

(21)出願番号　特願平10－52971
(22)出願日　平成10年２月18日(1998.2.18)
(65)公開番号　特開平11－228245
(43)公開日　平成11年８月24日(1999.8.24)
審査請求日　平成11年８月４日(1999.8.4)
前置審査

(73)特許権者　000004064
日本碍子株式会社
愛知県名古屋市瑞穂区須田町２番56号
(72)発明者　牧野　琢磨
愛知県名古屋市瑞穂区須田町２番56号
日本碍子株式会社内
(72)発明者　新海　正幸
愛知県名古屋市瑞穂区須田町２番56号
日本碍子株式会社内
(74)代理人　100088616
弁理士　渡邊　一平
審査官　近野　光知

最終頁に続く

(54)【発明の名称】　２種類以上の異種部材よりなる複合部材を製造する方法

1

(57)【特許請求の範囲】
【請求項１】　相互にその熱応力を異にする２種類以上の異種部材同士を接合させるのに充分な間隔を置いて互いに向かい合わせに配置させ、該間隔に所定量のセラミックまたはサーメット微粒子を充填し、引き続いて溶融状態にした所定量の硬ろう材を流し込み、次いで冷却して該２種類以上の異種部材同士を接合させることを特徴とする、２種類以上の異種部材よりなる複合部材を製造する方法。
【請求項２】　該セラミック微粒子が金属でメッキまたは被覆されたものである、請求項１に記載の２種類以上の異種部材よりなる複合部材を製造する方法。
【請求項３】　冷却を徐冷方法により行うものである、請求項１または２に記載の２種類以上の異種部材よりなる複合部材を製造する方法。

2

【請求項４】　硬ろう材がベース金属としてＡｕ、Ａｇ、Ｃｕ、Ｐｄ、ＡｌまたはＮｉを含むものである、請求項１～３のいずれか１項に記載の２種類以上の異種部材よりなる複合部材を製造する方法。
【請求項５】　該２種類以上の異種部材の少なくとも一つがセラミック製部材である請求項１～４のいずれか１項に記載の２種類以上の異種部材よりなる複合部材を製造する方法。
【請求項６】　該２種類以上の異種部材が金属製部材とセラミック製部材との組み合わせである請求項５に記載の２種類以上の異種部材よりなる複合部材を製造する方法。
【請求項７】　該複合部材がガス分離用部材である請求項１～６のいずれか１項に記載の２種類以上の異種部材よりなる複合部材を製造する方法。

3

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】　本発明は、相互にその熱応力を異にする２種類以上の異種部材同士を接合させて、該２種類以上の異種部材からなる複合部材を製造する方法に関する。

【０００２】

【従来の技術】　異種部材同士、特に、一方の部材が熱応力に対して脆弱な性質を有する場合には、該異種部材同士の接合工程、特に、高温での接合後の冷却操作中に接合界面近傍でクラックが生じて所望の接合強度を保持できなくなりかくして生産された異種部材同士の接合体である複合部材が使用中に破損して事故を起す等の問題が生じたり、熱応力に弱い部材にクラックが生じて、複合部材としての高気密性を保持できなくなり製造時に不良品として処分せざるを得なくなったりするために、結果としてこの様な複合部材が全体として高コストとなるなどの不都合が生じている。例えば、異種部材同士の接合例であるセラミック製部材と金属製部材との接合には、ろう材を用いる方法があるが、この場合には、セラミック製部材とろう材との濡れを確保するためにセラミック製部材の接合面の表面を金属、例えば、Ｎｉ等の金属でメッキした後、両部材を適当な間隔をおいて向かい合わせて配置させ、この間隔にろう材を流し込み、接合させる方法が通常採用されている。しかしながらこの方法では、熱応力を低下させるのには、充分でなく、熱応力に対して脆弱なセラミック製部材側にしばしばクラックが形成され、結合強度ばかりでなく複合部材として要求される気密性などの各種性能に影響を及ぼすので好ましくない。また、熱応力を緩和する方法としては、接合の際に熱膨張率の低い金属を中間材として使用する方法、セラミックとの反応性に富み、塑性変形することにより応力を緩和することのできる軟質金属を中間材として使用する方法などが採用されたりしている。しかしこれらの技術も何れも一長一短があり、必ずしも汎用性の高い技術とは言えない。また、現在開発中の技術として高圧固相接合法があるが、まだ、実用化するには未解決の課題があり、従って、この方法では充分な結合強度が出ていないのが現状である。一方、複合半田としては、半導体チップと基板との固着に使用するものであって、半田よりも融点の高い材質からなる粉末体を混合したものが特開平６－１２６４７９号公報に開示されているが、この複合半田は、半田本体の中央部にのみ半田よりも融点の高い材質からなる粉末体を充填させることにより、従来の複合半田が有している表面にも存在している粉末体に起因する半田濡れ不良を解消すること、換言すれば、接合界面での接合強度を増加させることを目的とするものであるが、しかし、この複合半田は、熱応力の低下には有効ではなく、従って、熱応力に対する強度が著しく異なる異種部材同士の接合には使用できない。

4

【０００３】

【発明が解決しようとする課題】　本発明は、異種部材同士を適度な結合強度を保持しながら、高温での接合後の冷却操作の間における熱応力による接合界面近傍での接合強度の低下現象や、熱応力に対して弱い部材での冷却操作中におけるクラックの発生のない２種以上の異種部材からなる複合部材を製造する方法を提供せんとするものである。

【０００４】

【課題を解決するための手段】　本発明者等は、上記の様な現状に鑑みて種々検討した結果、接合部材の種類や形状等による制約が少なく、接合形状も選択の余地の多い硬ろう材をベースとして用いること、この硬ろう材に熱応力を低下させる微粒子状の物質を添加することにより、上記の目的が達成できることを見いだし本願発明を完成したものである。

【０００５】

【発明の実施の形態】　本発明に係る２種類以上の異種部材同士を接合させて２種類以上の異種部材よりなる複合部材を製造する方法は、先ず相互にその熱応力を異にする２種類以上の異種部材同士を接合させるのに充分な間隔を置いて互いに向かい合わせに配置させ、該間隔に所定量のセラミックまたはサーメット微粒子を充填し、引き続いて溶融状態にした所定量の硬ろう材を流し込み、次いで冷却して該２種類以上の異種部材同士を接合させて、２種類以上の異種部材よりなる複合部材を製造することからなる。本発明に係る２種類以上の異種部材よりなる複合部材を製造する方法において使用可能な硬ろう材としては、セラミック製部材同士、金属製部材同士、またはセラミック製部材と金属製部材といった熱応力を含む各種特性の異なる異種部材同士を接合するのに使用できるものであれば、その種類を問わず使用できる。本発明に係る２種類以上の異種部材よりなる複合部材を製造する方法において使用する硬ろう材としては、Ａｕ、Ａｇ、Ｃｕ、Ｐｄなどの貴金属をベースとしたもの、Ａｌ、Ｎｉなどの金属をベースとした汎用性硬ろう材が挙げられる。勿論、接合する部材の性質との関係でより適切なものを選択すれば良い。接合する部材のいずれかがセラミック製部材の場合、特に、多孔質性のセラミック部材のときは、貴金属元素、例えば、Ａｕ、Ａｇ、Ｃｕ等をベースとした硬ろう材が好適に使用される。このような硬ろう材としては、Ａｇ－Ｃｕ－Ｔｉ系硬ろう材、Ａｇ－Ｃｕ系硬ろう材などが挙げられる。中でも、２７％のＣｕを含む銀ろう系の硬ろう材であるＢＡｇ－８（熔融点：７８０℃）等が好適に使用される。熱応力を低下させる微粒子状の物質としては、セラミック製の微粒子、セラミックと金属との複合材料であるサーメット微粒子、低熱膨張性金属微粒子などが好適に使用される。セラミック微粒子としては、窒化珪素、窒化アルミニウム、アルミナ、炭化珪素などの微粒子が挙げ

られる。サーメット微粒子としては、Ｎｉ－Al_2O_3、Ｃｕ－Al_2O_3等の微粒子が挙げられる。低熱膨張性金属微粒子としては、高温での熱膨張率の低いモリブデン、タングステン等の金属の微粒子が挙げられる。熱応力を効率よく低下させるためには、これらの物質の平均粒度を一定の範囲内にすることが必要となる。好ましい平均粒度は、１マイクロメーターから１００マイクロメーターである。平均粒径の異なる２種類以上の微粒子を混合して使用しても勿論差し支えない。セラミックを使用する場合には、硬ろう材との濡れが問題となるので、表面を金属、例えば、Ｎｉ、Ｃｕ、Ａｇ等の金属でメッキするか、またはＡｕ、Ａｇ、Ｔｉなどをスパッタすることにより被覆する必要がある。メッキ方法としては特に制限はないが、無電解メッキなどが好適に使用される。硬ろう材と熱応力を低下させる微粒子状の物質とは、通常、使用前に７０：３０から１０：９０の比率で混合して使用すれば良い。そして、熱応力を低下させる微粒子状の物質を予め所定量接合箇所に充填しておき、それに所定量の溶融状態の硬ろう材を流し込んで接合する。

【０００６】　本発明に係る２種類以上の異種部材よりなる複合部材を製造する方法により接合できる異種部材の組み合わせとしては、セラミック製の部材と金属製部材、諸性質を異にするセラミック製部材同士、諸性質を異にする金属製の部材同士が挙げられる。セラミック部材としては、非酸化物系のセラミック部材、酸化物系のセラミック部材の両者に使用できるが、熱膨張係数のより少ない非酸化物系セラミック部材と他の部材との接合に好適に使用できる。係る接合例としては、セラミック製ガス分離用部材と金属製の口金部材等の例を挙げることができる。本願発明に係る２種類以上の異種部材よりなる複合部材を製造する方法によれば、接合時の８００℃またはそれ以上の高温から常温（２５℃前後）迄品温を下げてゆく操作の間における熱応力の発生を著しく低下させることが出来るので、著しく熱応力のことなる異種部材同士の接合、例えば、熱膨張係数の低い非酸化物系のセラミック製部材と熱膨張率の高い金属製部材の接合において所望の効果を発揮することが出来るのである。

【０００７】　本発明に係る２種類以上の異種部材よりなる複合部材を製造する方法による２種類以上の異種部材の接合は、該間隔に熱応力を低下させる物質を予め所定量のセラミックまたはサーメット微粒子を充填しておき、ついで、硬ろう材をその熔融点以上に加熱し溶融させた後、これを接合箇所に所定量流し込んで、接合すれば良く、その後常法に従い冷却操作を行えば良い。冷却時間は、接合する異種部材の特性などを考慮して定めれば良いが、通常は、１時間から１０時間の範囲内である。冷却操作の際、徐冷方法を採用すると、熱応力の影響を著しく低下させることが出来るのでより好ましい。

なお、徐冷方法とは、通常の冷却方法に掛ける時間の約２倍またはそれ以上の時間を掛けて冷却することをいい、接合部への熱応力の影響を最小限にすることができる。勿論、使用する硬ろう材の成分や、接合する部材の種類により徐冷に必要な時間は異なるので、予め小規模実験により、最適な徐冷時間を定めることが好ましい。本願発明に係る２種類以上の異種部材よりなる複合部材を製造する方法により製造される複合部材としては、２種類以上の異種部材同士を接合したもの、例えば、多孔質性のセラミック製部材と熱膨張係数の著しく高い金属製部材とを接合して得られた複合部材や、熱膨張係数が相互に異なるセラミック製部材同士、または、熱膨張率が異なる金属製部材同士を接合して得られる複合部材が挙げられる。より具体的には、ガスの分離に使用される多孔質性アルミナ製部材と各種ガス分析装置に装着するための金属製の金口部材を接合して形成したガス分離用複合部材等が挙げられる。勿論、３種類またはそれ以上の異種部材同士を接合したものも本発明で言うところの複合部材に含まれることは言うまでもない。

【０００８】

【実施例】　以下実施例と比較例とを挙げて、本発明を説明するが、勿論、本発明は、これらの例により何等制限されるものではないことは言うまでもない。なお、残留応力の大きさには分布があり、それを直接測定するのは困難であるため、以下の方法で歪量を測定し、それをもって応力値を算出し、残留応力の目安とした。この残留応力の測定は図１で示したように、接合材３を介して金属製部材１とセラミック製部材２を接合した接合体のセラミック製部材のほぼ中央に歪ゲージを貼り付けた後、金属製部材を強制的に取り除き、冷却操作の際生じた歪量を歪ゲージにより測定することにより行う。

（実施例）

セラミック製部材としてアルミナ製ガス分離用部材と金属製部材としての装着用の金口部材とを、熱応力を低下させる微粒状の物質として平均粒径４０マイクロメーターのアルミナ微粒子をＮｉメッキさせたものをＢＡｇ－８硬ろう材に４０：６０の割合で添加して調製した接合材を用いて接合した。接合の条件は以下の通りである。

接合温度：８００℃

保持時間：１０分間（８００℃）

昇降温速度：８００℃／時間

（室温から８００℃まで１時間、８００℃から室温まで１時間）

雰囲気：真空（10^{-6} torr）

同時に、同じ材料を用いて、５時間掛けて、徐冷操作により調製したものも用意した。かくして調製した２種類の複合部材について、上記の方法により残存応力を測定したところ、それぞれ、６７．３および１７．６ＭＰａであった。また、常法に従い調製した複合部材の接合部の界面の状態を電子顕微鏡で調査したところ、図２に示

したように全くクラックの発生は認められなかった。

（比較例）

上記の実施例で使用したものと同一のアルミナ製ガス分離用部材と金属製部材との接合を硬ろう材ＢＡｇ－８を用いて行った。なお、接合に際して、アルミナ製ガス分離用部材の接合面はＮｉメッキを施した。常法に従い冷却操作後、残存応力を上記の方法で調査したところ１０５ＭＰａであった。また、この複合部材の接合部の界面の状態を電子顕微鏡で調査したところ図３に示すようなクラックの発生が認められた。

【０００９】

【発明の効果】　本願発明に係る２種類以上の異種部材よりなる複合部材を製造する方法によれば、残留応力が少なく、そのために機械的破壊に弱いセラミックなどの

非金属製部材側でのクラックの発生もなく、充分な接合強度を有する複合部材を製造することができる。また、クラックの発生がないため高度の気密性が求められる複合部材として優れたものを提供できると言う効果もある。

【図面の簡単な説明】

【図１】　残留応力の測定に使用する複合部材の構成を示す模式図である。

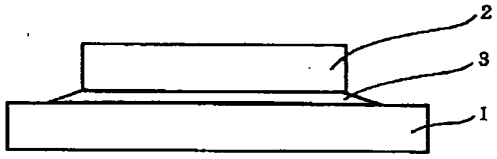
【図２】　本発明に係る複合部材の接合部の界面の粒子構造を示す電子顕微鏡写真である。

【図３】　従来の方法に係る複合部材の接合部の界面の粒子構造を示す電子顕微鏡写真である。

【符号の説明】

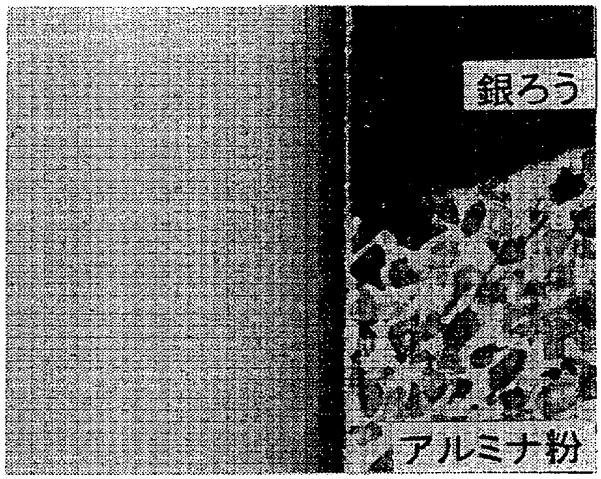
１…金属製部材、２…セラミック製部材、３…接合材。

【図１】



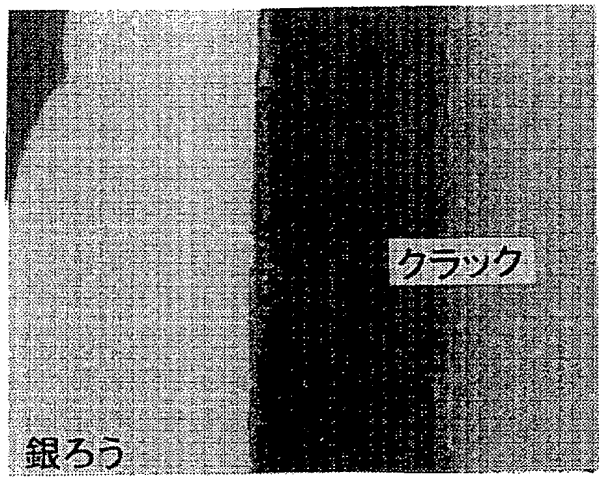
【図２】

図面代用写真



【図３】

図面代用写真



フロントページの続き

(56)参考文献　特開　平５－319944（ＪＰ，Ａ）
特開　平５－246769（ＪＰ，Ａ）
特開　平３－205389（ＪＰ，Ａ）
特開　平３－80160（ＪＰ，Ａ）
特開　平２－202041（ＪＰ，Ａ）
特開　平１－249296（ＪＰ，Ａ）
特開　平７－69749（ＪＰ，Ａ）
特開　平５－163078（ＪＰ，Ａ）
特開　昭62－179889（ＪＰ，Ａ）
特開　昭63－8273（ＪＰ，Ａ）
特開　平２－38376（ＪＰ，Ａ）

JAPANESE [JP,11-228245,A]

CLAIMS DETAILED DESCRIPTION TECHNICAL FIELD PRIOR ART EFFECT OF THE INVENTION TECHNICAL PROBLEM MEANS EXAMPLE DESCRIPTION OF DRAWINGS DRAWINGS

[Translation done.]

* NOTICES *

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

CLAIMS

[Claim(s)]
[Claim 1] An adhesives constituent for joining at least two kinds of different-species members which consists of matter and brazing solder material of the shape of a particle to which thermal stress is reduced.
[Claim 2] A constituent whose matter of the shape of this particle it is the brazing solder material whose base metal of this brazing solder material is Au, Ag, Cu, Pd, aluminum, or nickel in a constituent according to claim 1, and is a ceramic particle, a cermet particle, or a low expansion metal particle.
[Claim 3] A constituent the front face of whose is the ceramic particle by which matter of the shape of this particle was covered with plating or a spatter with a metal in a constituent according to claim 1 or 2.
[Claim 4] two or more kinds of different-species members which differ in the thermal stress mutually -- this -- a compound member which becomes any 1 term of claims 1-3 which have joined mutually two or more kinds of different-species members from a constituent of a publication.
[Claim 5] this -- a compound member according to claim 4 whose at least one of two or more kinds of the different-species members is a member made from a ceramic.
[Claim 6] this -- a compound member according to claim 4 or 5 two or more kinds of whose different-species members are the combination of a metal member and a member made from a ceramic.
[Claim 7] A compound member given in any 1 term of claims 4-6 these whose compound members are members for gas separation.
[Claim 8] sufficient gap to join mutually two or more kinds of different-species members which differ in the thermal stress is kept, and it arranges face to face mutually -- making -- a constituent given in this gap at any 1 term of claims 1-3 -- slushing -- subsequently -- cooling -- this -- a method of joining two or more kinds of different-species members, and manufacturing a compound member.
[Claim 9] A way according to claim 8 this compound member consists of a member made from a ceramic, and a metal member.
[Claim 10] brazing solder material of the specified quantity which kept sufficient gap to join mutually two or more kinds of different-species members which differ in the thermal stress, was arranged face to face mutually, filled up this gap with a ceramic or a cermet particle of the

specified quantity, and was succeedingly changed into a melting condition -- slushing -- subsequently -- cooling -- this -- a method of joining two or more kinds of different-species members, and manufacturing a compound member.

[Translation done.]

JAPANESE [JP,11-228245,A]

CLAIMS DETAILED DESCRIPTION TECHNICAL FIELD PRIOR ART EFFECT OF THE INVENTION TECHNICAL PROBLEM MEANS EXAMPLE DESCRIPTION OF DRAWINGS DRAWINGS

[Translation done.]

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[The technical field to which invention belongs] This invention relates to utilization of the adhesives constituent with which residual stress was reduced remarkably, and this constituent. Furthermore, it is related with the compound member joined to joining two or more sorts of members from which coefficient of thermal expansion differs remarkably mutually in detail, maintaining high airtightness using the usable adhesives constituent and this adhesives constituent, and the method of manufacturing a compound member using this adhesives constituent.
[0002]
[Description of the Prior Art] Different-species members and when one member has a brittle property to thermal stress especially Problems, such as damaging, while the compound member which is the zygote of the different-species members which the crack arose near the cementation interface, and it becomes impossible to have held desired bonding strength, and were produced in this way during the cooling actuation after the cementation especially in an elevated temperature uses it, the cementation process of these different-species members and, and causing accident, arise, or The crack arose in the member weak to thermal stress, and since it becomes impossible to hold the high airtightness as a compound member and cannot but stop disposing as a defective at the time of manufacture, inconvenience, like such a compound member serves as high cost as a whole as a result has arisen. For example, although there is a method of using wax material in cementation to the member made from a ceramic and metal member which are the example of cementation of different-species members The front face of the plane of composition of the member made from a ceramic in order to secure **** of the member made from a ceramic, and wax material In this case, a metal, For example, after plating with metals, such as nickel, both members are set, and a suitable gap is opposed and is arranged, wax material is slushed into this gap and the method to which it is made to join is usually adopted. However, this method is not enough to reduce thermal stress, and since a crack is often formed in the brittle member side made from a ceramic to thermal stress and various engine performance, such as not only bond strength but airtightness demanded as a compound member, is affected, it is not desirable. Moreover, the method of using the elasticity metal which can ease stress as medium material etc. is adopted by being rich in reactivity with the method and ceramic which are used as medium material, and deforming plastically a metal with a low coefficient of thermal expansion as a method

of easing thermal stress, in the case of cementation. However, each of such technology also has merits and demerits, and cannot necessarily be said to be the high technology of versatility. Moreover, although there is a high voltage solid-state-welding method as technology under current development, the actual condition is that there is still an unsolved technical problem in putting in practical use, therefore bond strength sufficient by this method has not come out. On the other hand, as cored solder, use it for fixing with a semiconductor chip and a substrate, and although what mixed the powder object which consists of construction material with the melting point higher than solder is indicated by JP,6-126479,A This cored solder by making only the center section of the solder main part fill up with the powder object which consists of construction material with the melting point higher than solder Although it aims at canceling poor solder **** resulting from the powder object which exists also in the front face which the conventional cored solder has, and making the bonding strength in a cementation interface increase if it puts in another way However, this cored solder is not effective in lowering of thermal stress, therefore reinforcement to thermal stress cannot use it for cementation of remarkably different different-species members.

[0003]

[Problem(s) to be Solved by the Invention] The lowering phenomenon of the bonding strength near [while this invention holds moderate bond strength for different-species members] the cementation interface by the thermal stress between the cooling actuation after the cementation in an elevated temperature, Let the method of manufacturing the compound member joined using the glue constituent between two or more sorts of different-species members which do not have generating of the crack under cooling actuation by the weak member to thermal stress, and this adhesives constituent, and the compound member using this adhesives constituent be an offer plug.

[0004]

[Means for Solving the Problem] As a result of examining many things in view of the above actual condition, this invention person etc. has little constraint by a class, a configuration, etc. of joint material, that a cementation configuration also uses brazing solder material with much room of selection as the base, and by adding matter of the shape of a particle which reduces thermal stress to this brazing solder material, finds out that the above-mentioned object can be attained and completes the invention in this application.

[0005]

[Embodiment of the Invention] The adhesives constituent concerning this invention usable to cementation of two or more sorts of different-species members can be manufactured by adding the matter of the shape of a particle which reduces thermal stress to brazing solder material. If it can be used for joining the members made from a ceramic, metal members, or the different-species members from which the various properties containing the thermal stress called the member made from a ceramic and metal member differ as brazing solder material, it can be used regardless of the class. The versatility brazing solder material which used as the base metals which used noble metals, such as Au, Ag, Cu, and Pd, as the base, such as a thing, and aluminum, nickel, as brazing solder material used for the adhesives constituent concerning this invention is mentioned. Of course, what is necessary is just to choose a more suitable thing by relation with the property of the member to join. Especially when either of the members to join is a

member made from a ceramic, and it is the ceramic member of porosity nature, the brazing solder material which used the noble-metals element, for example, Au, Ag, Cu, etc., as the base is used suitably. As such brazing solder material, Ag-Cu-Ti system brazing solder material, Ag-Cu system brazing solder material, etc. are mentioned. Especially, BAg-8 (a fusing point: 780 degrees C) which is the brazing solder material of the silver solder system containing 27% of Cu is used suitably. As matter of the shape of a particle to which thermal stress is reduced, the particle made from a ceramic, the cermet particle which is the composite material of a ceramic and a metal, a low thermal-expansion nature metal particle, etc. are used suitably. As a ceramic particle, particles, such as silicon nitride, alumimium nitride, an alumina, and silicon carbide, are mentioned. As a cermet particle, the particle of nickel-aluminum 2O3 and Cu-aluminum2O3 grade is mentioned. The particle of metals, such as molybdenum with a coefficient of thermal expansion low as a low thermal-expansion nature metal particle in an elevated temperature and a tungsten, is mentioned. In order to reduce thermal stress efficiently, it is necessary to carry out average grain size of these matter within fixed limits. A desirable average grain size is 100 micrometers from 1 micrometer. Even if it mixes and uses two or more kinds of particles from which mean particle diameter differs, of course, it does not interfere. Since **** with brazing solder material poses a problem in using a ceramic, it is necessary to cover by plating a front face with a metal, for example, metals, such as nickel, Cu, and Ag, or carrying out the spatter of Au, Ag, Ti, etc. Although there is especially no limit as the plating method, electroless deposition etc. is used suitably. Although what is necessary is just to usually use it before an activity, mixing by the ratio of 70:30 to 10:90, brazing solder material and the matter of the shape of a particle to which thermal stress is reduced fill up the specified quantity cementation part with the matter of the shape of a particle to which thermal stress is reduced beforehand, may slush the brazing solder material of the melting condition of the specified quantity into it, and may join it to it.
[0006] As a combination of a different-species member joinable [with the adhesives constituent concerning this invention], the member made from a ceramic, a metal member, the members made from a ceramic that differ in many properties, and the metal members which differ in many properties are mentioned. As a ceramic member, although it can be used for both ceramic member of a non-oxide system, and ceramic member of an oxide system, it can be used suitable for cementation to fewer non-oxide system ceramic members of a coefficient of thermal expansion, and other members. the mouthpiece of the member for the gas separation made from a ceramic and metal as a starting example of cementation -- examples, such as a member, can be given. since generating of the thermal stress between the actuation which lowers the temperature of goods from the elevated temperature beyond 800 degrees C or it at the time of cementation to ordinary temperature (before or after 25 degrees C) can be reduced remarkably in the case of the adhesives constituent concerning the invention in this application -- remarkable -- thermal stress -- things -- the desired effect can be demonstrated in cementation of different-species members, for example, cementation of the member made from a ceramic of a non-oxide system with a low coefficient of thermal expansion, and a metal member with a high coefficient of thermal expansion.

[0007] The cementation method concerning this invention changes into a melting condition the adhesives constituent which is the mixture of the brazing solder material concerning this invention,

and the matter of the shape of a particle to which thermal stress is reduced, slushes it into the joint between at least two or more sorts of different-species members, and should just perform cooling actuation according to a conventional method after that. Although what is necessary is just to define a cooldown delay in consideration of the property of the different-species member to join etc., it is usually within the limits of 1 to 10 hours. If the annealing method is adopted in the case of cooling actuation, since the effect of thermal stress can be reduced remarkably, it is more desirable. In addition, the annealing method can mean cooling over the time amount beyond the twice [about] or it of time amount spent on the usual cooling method, and effect of the thermal stress to a joint can be made into the minimum. Of course, since time amount required for annealing changes with the component in the adhesives constituent to be used, and classes of member to join, it is desirable to define the optimal annealing time amount by the bench scale test beforehand. Of course, the cementation part of specified quantity different-species members is beforehand filled up with the matter to which thermal stress is reduced, and after that, after it heats brazing solder material beyond the fusing point and it carries out melting, this is made into a cementation part in the style of the specified quantity, and you may join. The cooling method is the same as the case of the adhesives constituent which is made to mix brazing solder material and the matter of the shape of a particle to which thermal stress is reduced, and is obtained. What joined two or more kinds of different-species members with the adhesives constituent concerning the invention in this application as a compound member concerning the invention in this application, for example, the compound member which joined the member made from a ceramic of porosity nature and the high metal member with a remarkable coefficient of thermal expansion, and was obtained, and the compound member which joins the members made from a ceramic from which a coefficient of thermal expansion differs mutually, or the metal members from which coefficient of thermal expansion differs, and is obtained mention, and it is ********. The compound member for gas separation which joined and formed the metal golden regio-oralis material for more specifically equipping the member made from a porosity nature alumina and the various gas analysers which are used for separation of gas is mentioned. Of course, it cannot be overemphasized that it is contained in the compound member which says by this invention what joined three kinds or the different-species members beyond it.

[0008]

[Example] Although an example and the example of a comparison are given below and this invention is explained, of course, it cannot be overemphasized that this invention is not what is restricted in any way by these examples. In addition, the magnitude of residual stress had distribution, and since it was difficult, measuring it directly measured the deformation amount by the following methods, it computed the stress value with it, and was taken as the rule of thumb of residual stress. As drawing 1 showed, after [the member made from a ceramic of the zygote which joined the metal member 1 and the member 2 made from a ceramic through the adhesives constituent 3] sticking a strain gage in the center mostly, measurement of this residual stress removes a metal member compulsorily, and is performed by measuring the deformation amount produced at the time of cooling actuation by the strain gage.

(Example) It joined using the adhesives constituent concerning the invention in this application which added and prepared at a rate of 40:60 what carried out nickel plating of the alumina particle

with a mean particle diameter of 40 micrometers as microparticulate matter to which the member for the gas separation made from an alumina and the golden regio-oralis material for wearing as a metal member reduce thermal stress as a member made from a ceramic to BAg-8 brazing-solder material. The conditions of cementation are as follows.

virtual-junction-temperature: -- 800-degree-C holding-time: -- for 10 minutes (800 degrees C)

Rising-and-falling-temperature speed : 800 degrees C (1 hour from 1 hour and 800 degrees C to [from a room temperature to 800 degrees C] a room temperature)/hour

Ambient atmosphere: Vacuum (10-6torr)

Simultaneously, what was prepared by annealing actuation was prepared over 5 hours using the same material. When residual stress was measured by the above-mentioned method and having been carried out about the compound member of two kinds of having prepared in this way, they were 67.3 and 17.6MPa(s), respectively. Moreover, when the condition of the interface of the joint of the compound member prepared according to the conventional method was investigated with the electron microscope, as shown in drawing 2 , generating of a crack was not accepted at all.

(Example of a comparison) cementation to the same member for the gas separation made from an alumina as what was used in the above-mentioned example, and a metal member -- brazing solder material -- it carried out using BAg-8. In addition, on the occasion of cementation, the plane of composition of the member for the gas separation made from an alumina performed nickel plating. It was 105MPa when residual stress was investigated by the above-mentioned method after cooling actuation according to the conventional method. Moreover, when the condition of the interface of the joint of this compound member was investigated with the electron microscope, generating of a crack as shown in drawing 3 was accepted.

[0009]

[Effect of the Invention] The compound member in which residual stress does not have generating of the crack by the side of members made from a nonmetal, such as a weak ceramic, in mechanical destruction few therefore, either, and the adhesives constituent concerning the invention in this application has sufficient bonding strength can be manufactured. Moreover, since there is no generating of a crack, there is an effect referred to as being able to offer what was excellent as a compound member asked for advanced airtightness.

[Translation done.]

JAPANESE [JP,11-228245,A]

CLAIMS DETAILED DESCRIPTION TECHNICAL FIELD PRIOR ART EFFECT OF THE INVENTION TECHNICAL PROBLEM MEANS EXAMPLE DESCRIPTION OF DRAWINGS DRAWINGS

[Translation done.]

* NOTICES *

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]
[Drawing 1] It is the mimetic diagram showing the configuration of the compound member used for measurement of residual stress.
[Drawing 2] It is the electron microscope photograph in which the particulate structure of the interface of the joint of the compound member concerning this invention is shown.
[Drawing 3] It is the electron microscope photograph in which the particulate structure of the interface of the joint of the compound member concerning the conventional method is shown.
[Description of Notations]
1 -- A metal member, 2 -- The member made from a ceramic, 3 -- Adhesives constituent.

[Translation done.]